

Synthesis of high-purity Ti_2SC powder by microwave hybrid heating

Chunlong GUAN, Nana SUN*

School of Material Science and Engineering, Henan University of Technology, Engineering Laboratory of High Temperature Resistance-Wear Materials, Zhengzhou 450007, China

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Abstract: A novel simple method is presented to synthesize high-purity Ti_2SC powder using Ti/C/S and Ti/C/ TiS_2 systems by microwave hybrid heating at different temperatures in argon atmosphere. It was confirmed that the synthesis temperature is strongly dependent on the starting composition. For Ti/C/S system, Ti_2SC with small amounts of TiS and TiC was synthesized at 1200 °C. For Ti/C/ TiS_2 system, high-purity Ti_2SC was synthesized at 800 °C and above. The synthesis of Ti_2SC powder at low temperature was attributed to the combination of microwave effect by microwave hybrid heating and the introduction of TiS_2 as sulfur source. Scanning electron microscopy (SEM) analysis indicated that the layered structure of Ti_2SC particles is perfectly formed at 1100 °C, and the crystal particle size approaches to homogeneity which is about 2–5 μm. It was presumed that the formation mechanism of Ti/C/ TiS_2 system is that TiS_2 firstly reacts with Ti to form Ti–S intermetallics, then Ti–S intermetallics reacts with un-reacted Ti and graphite to produce Ti_2SC .

Keywords: Ti_2SC ; microwave hybrid heating; formation mechanism

1 Introduction

$\text{M}_{n+1}\text{AX}_n$ phases (where $n=1, 2, 3$, M is an early transition metal, A is a IIIA or IVA element, and X is C or N, abbreviated as MAX) are lately of higher interest because of their unique properties of ceramics and metals [1–3], such as readily machinable but high elastic stiffness, high thermal and electrical conductivity, excellent thermal shock resistance and damage tolerance [4–10], which are crucial for their potential application.

Ti_2SC , as a member of S-containing MAX phases, has the lowest c/a ratio of all MAX phases [11]. So it is reasonable to believe that Ti_2SC would exhibit unusual mechanical and physical properties. Very recent results confirmed this notion [12]. For example, its Vickers

hardness (8 ± 2 GPa) is significantly higher than that of the vast majority of MAX phases. Furthermore, its Young's modulus is one of the highest among that of MAX phases measured to date [13]. Ti_2SC also exhibits stable structure under about 50 GPa [14], and good thermal stability at high temperature [15] that is resulted from its strong Ti–S hybridization, further revealing that it could be a promising candidate for high-temperature application. In addition, its superior tribological properties against alumina at 26–550 °C and self-lubricity [16] make this material potential to be applied on machinery, transportation, and so on.

So far, bulk Ti_2SC ceramic is obtained by hot pressing [12–17] and spark plasma sintering [18] generally. Due to most of bulk Ti_2SC ceramic sintered using Ti_2SC powder [12–16], the synthesis of high-purity Ti_2SC powder becomes quite necessary. However, the synthesis of high-purity Ti_2SC powder was rarely reported. Recently, Chen *et al.* [19] utilized iron

* Corresponding author.

E-mail: cailiaosunna@163.com

disulfide or iron sulfide to synthesize Ti_2SC at 1500 °C in argon by pulse-electric-current-aided. Zhu *et al.* [17] prepared Ti_2SC powder by pressureless heating a mixture of TiS_2 , Ti, and C at 1600 °C for 2 h in argon. Li *et al.* [20] and Liang *et al.* [21] used S, Ti, and C as starting materials to synthesize Ti_2SC powder at 2125 and 2410 °C by combustion synthesis, respectively. The results showed that a large amount of impurities such as TiC and Ti–S intermetallics exist in the final products, and the synthesis temperature of Ti_2SC is above 1500 °C. It is clear that these methods require considerable time, consume a lot of energy, and contaminate the environment.

Recently, microwave heating method, as a new route, could well supply the homogeneous heating in the sintered materials during the volumetric heating process, which has aroused researchers' interest because of its advantages, such as rapid heating rate, uniform heating, selective energy absorption, and simple operating. Presently, high-purity Ti_3SiC_2 and Ti_3AlC_2 powders have been successfully synthesized by microwave heating [22,23]. Hence, it is reasonable to expect that the microwave heating method can provide a new approach to fabricate high-purity Ti_2SC powder.

In this work, microwave hybrid heating was introduced to synthesize highly pure Ti_2SC powder using Ti/C/S and Ti/C/ TiS_2 systems. The reaction mechanism and the optimization of synthesis process of pure Ti_2SC at low temperature were studied.

2 Experimental procedure

Powders of Ti (99%, 200 mesh), S (99.5%, 100 mesh), and graphite (98%, 100 mesh) were used as starting materials. Firstly, TiS_2 was obtained by heating the mixture of 1Ti:2.05S in the vacuum borax glass tube at 600 °C for 12 h. Then, S, Ti, and C powders with molar ratio of (1–1.2):2:1, or the as-received TiS_2 , Ti, and C powders with molar ratio of (1–1.3):(2.7–3):2, were mixed and compacted using a cylindrical steel die (8 mm in diameter). The compacts were put in a small alumina crucible covering by thermal insulation material to reduce the heat dissipation which was placed in the middle of large alumina crucible filled with microwave absorption material. The self-designed reactor was heated in a domestic microwave oven (equipment type BD1412, frequency 2.45 GHz, maximum power 3000 W) at 200–1100 °C (200 °C/min)

for 3–13 min in Ar atmosphere. Finally, the samples were cooled to room temperature under Ar atmosphere, and the powders were obtained by crushing the sintered block.

The phase composition of synthesis samples was identified with X-ray diffraction (XRD) experiments conducted on a Rigaku D/max-Ultima IV X-ray diffractometer using Cu $K\alpha$ radiation. The TOPAS 4.2 software developed by Bruker Corporation was selected for the Rietveld quantitative phase analysis (RQPA) of the obtained samples [24]. A scanning electron microscope (SEM, XL30 S-FEG, Japan) with energy dispersive spectroscopy (EDS) was used to investigate the morphology of synthesized Ti_2SC samples.

3 Results and discussion

3.1 Phase assemblage of the synthesized products

The elemental sulfur was first chosen as sulfur source to prepare Ti_2SC by microwave hybrid heating method. Considering the low boiling point of S (433.6 °C), severe evaporation of sulfur was foreseeable during heating. To make up the deficiency of sulfur, excess S was introduced in complementary experiments. Ti, S, and graphite powder mixtures with different molar ratios were microwave hybrid heated at 1100 °C for 3 min in flowing Ar. Phase compositions in the final products were determined by XRD, as shown in Fig. 1. The molar ratio is Ti:S:C=2:1:1 when the amount of the additive S is equal to 0 at%. It can be seen that TiC is the major phase in final products with a small amount of TiS_x , and only a few Ti_2SC phase appears. The amount

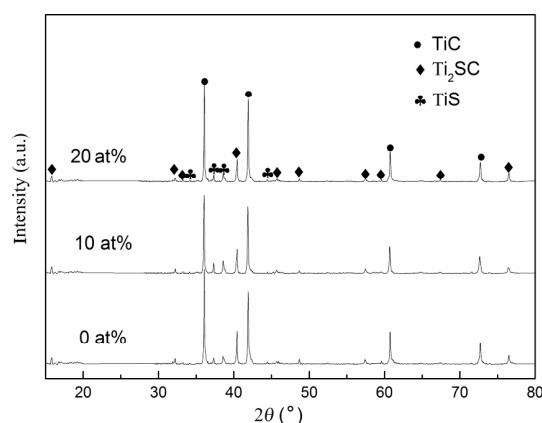


Fig. 1 XRD patterns of Ti/C/S mixtures with different amount of additive sulfur sintered by microwave hybrid heating at 1100 °C for 3 min in Ar atmosphere.

of Ti_2SC has no obvious change with increasing additive S from 10 to 20 at%. There exist the differences in comparison with the results reported by Li *et al.* [20], which suggested the increase of S content could promote the formation of Ti_2SC . Unfortunately, a large amount of TiC and Ti_3S_4 still exist in the final products.

Figure 2 reveals the XRD patterns of the Ti/C/S mixtures which were synthesized at various temperatures by microwave hybrid heating. At 1000 °C, the sample is mainly composed of un-reacted raw materials Ti and C, and little TiC and TiS exist. As temperature is increased to 1100 °C, there are large quantities of TiC, TiS, and un-reacted C, and a small amount of Ti_2SC is detected in the product. However, the contents of TiC and TiS impurities are reduced notably together with increase in the amount of Ti_2SC while sintering temperature rises. At calcining temperature of 1200 °C and above, small amounts of TiC and TiS impurities always exist, and Ti_2SC is determined as the predominant phase. By combustion synthesis using S, Ti, and graphite as starting materials, Ti_2SC with large amount of TiC and Ti_3S_4 was obtained at 2125 °C. Recently, the beneficial role of microwave on the low-temperature firing and reaction during microwave hybrid heating has been reported. Yadoji *et al.* [25] reported that microwave heating generates heat within the material first and then heats the entire volume. This heating mechanism is advantageous to rapid heating rate, uniform heating, high efficiency, and reduced cost [25,26]. The synthesis temperature and time decrease due to the effect of heating mechanism during microwave sintering. A detail synthesis mechanism of the Ti/C/S system will be discussed later.

XRD patterns illustrating the as-received TiS_2 and the

products of 3Ti/2C/1TiS₂ mixtures by microwave hybrid heating at different temperatures are shown in Fig. 3. It can be seen that TiS_2 is produced as a single phase in the final product. When the sample is heated at 1100 °C for 3 min, it is exciting that the purity of Ti_2SC improves considerably when substituting S with TiS_2 as sulfur source. However, when raising the temperature up to 1300 °C, the undesired TiC in the final product is still in a large proportion, indicating that the system is still under S-poor circumstance. Slightly excess of sulfur is introduced into the raw material in the complementary experiments.

Figure 4 shows the as-prepared samples with different amount of TiS_2 synthesized by microwave hybrid heating at 1100 °C for 3 min. As the amount of additive sulfur is 20 at%, the phase assemblage of sample consists of small amounts of un-reacted graphite, TiC, and other impurities. It is noted that the content of the impurities significantly decreases when compared

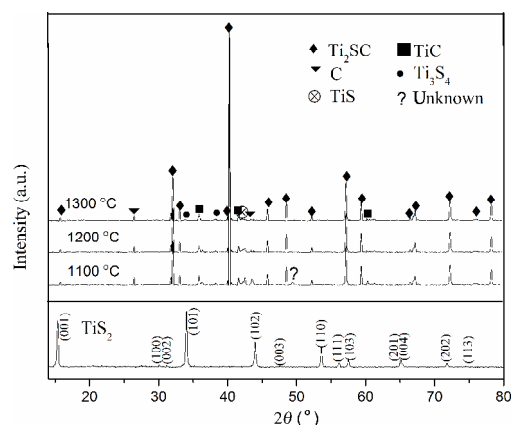


Fig. 3 XRD patterns of 3Ti/2C/1TiS₂ mixtures heated at different temperatures ranging from 1100 to 1300 °C and the as-received TiS_2 .

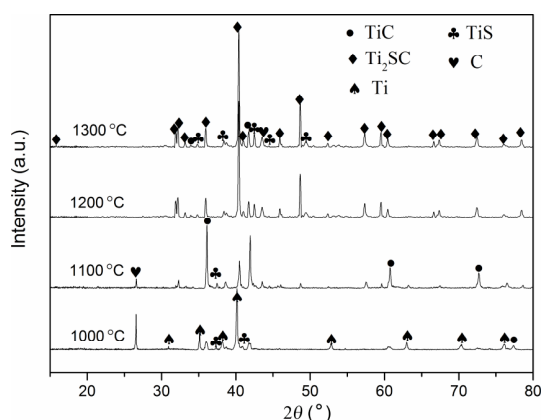


Fig. 2 XRD patterns of 2Ti/1C/1S mixtures heated at different temperatures ranging from 1000 to 1300 °C for 3 min in Ar atmosphere.

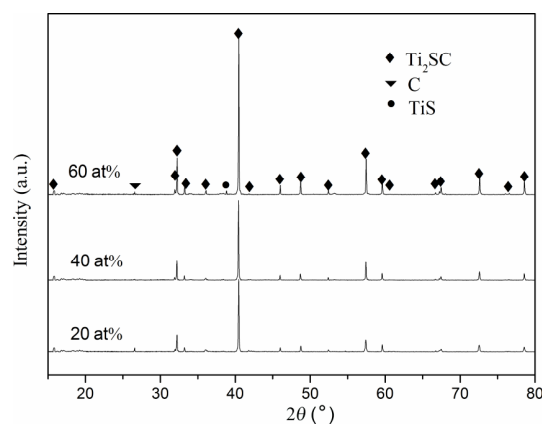


Fig. 4 XRD patterns of Ti/C/ TiS_2 mixtures with different amount of additive sulfur sintered at 1100 °C for 3 min in Ar atmosphere.

with that of the sample of 3Ti/2C/1TiS₂ (Fig. 3). When the amount of additive sulfur is equal to 40 at%, Ti₂SC becomes the only phase appearing in the sample. It is easy to see that the yield of Ti₂SC improves considerably using TiS₂ as sulfur source. Further increasing the amount of additive sulfur more than 40 at%, contributes little to the productivity of Ti₂SC. Based on the results mentioned above, further work focuses on the sample with the molar ratio of TiS₂:Ti:C = 1.2:2.8:2.

As can be seen in the diffractograms, shown in Fig. 5, the 2.8Ti/2C/1.2TiS₂ mixtures were synthesized by microwave hybrid heating at different temperatures in argon. At first stage, the sample heated below 300 °C is mainly composed of un-reacted starting materials Ti, TiS₂, and graphite with small amount of Ti–S intermetallic compounds (including Ti₅S₈, Ti₃S₄, and TiS). At 400 °C, in spite of existing a lot of un-reacted graphite, the most inspiring fact is that the desirable Ti₂SC has been produced as the major phase, while the amounts of Ti, TiS₂, and Ti₅S₈ compounds were disappeared completely. By further increasing in temperature, the amount of graphite reduces greatly and the quantity of Ti₂SC phase climbs rapidly. At 1100 °C, Ti_x (including Ti₅S₈, Ti₃S₄, TiS₂, TiS), Ti, and graphite are consumed completely, and Ti₂SC becomes the only phase in the sample. High-purity Ti₂SC is obtained at 1600 °C using traditional methods [16–18]. The decrease of synthesis temperature in this research is attributed to the combination of microwave effect by microwave hybrid heating [25,26] and the introduction of TiS₂ as sulfur source.

Figure 6 shows XRD patterns of 2.8Ti/2C/1.2TiS₂ mixtures heated at 1100 °C for different dwell time.

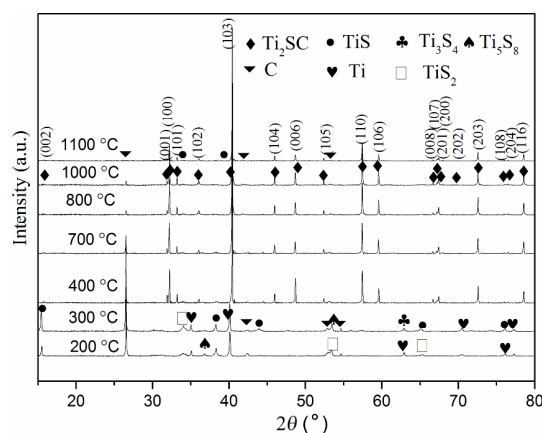


Fig. 5 XRD patterns of 2.8Ti/2C/1.2TiS₂ mixtures heated at different temperatures ranging from 200 to 1100 °C for 3 min in Ar atmosphere.

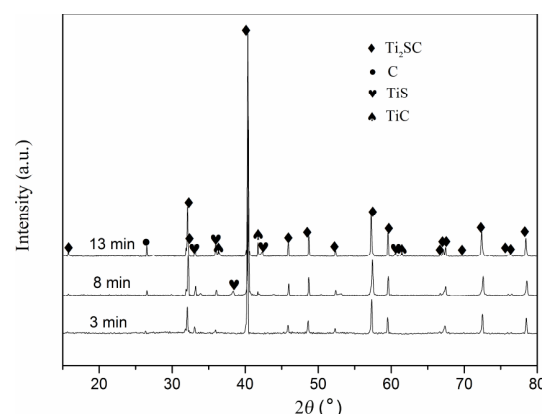


Fig. 6 XRD patterns of 2.8Ti/2C/1.2TiS₂ mixtures heated at 1100 °C for different dwell time.

When at 1100 °C for 3 min, Ti₂SC has been detected as the only phase in the sample. As the dwell time is prolonged further, TiC and TiS appear in the sample, implying that Ti₂SC decomposes under longer dwell time. Thus, the dwell time of 3 min is best for sintering of sample.

Figure 7 presents the Rietveld pattern of X-ray powder diffraction data for the sample 2.8Ti/2C/1.2TiS₂ mixture obtained by heating at 1100 °C for 3 min. As can be seen, visual results of the plot show the accomplishment of the Rietveld refinement, as reflected by small difference between the calculated and the observed. The calculated R_{wp} value is 5.083%, which is considered as acceptable according to basic principle of R_{wp} less than 20% [27]. High-purity Ti₂SC has indicated the presence of 97.48% of its phase with some minor phases TiS (0.97%) and C (1.55%) at 1100 °C.

When the starting composition and processing parameters are optimized, the content of Ti₂SC in the final products reaches 97.48 wt%, which is comparable to those pressure-assisted sintering samples [16,17].

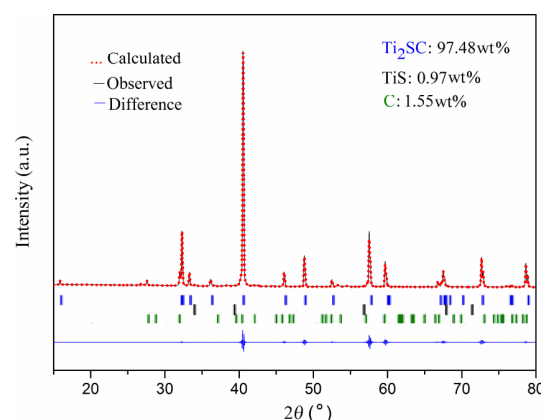


Fig. 7 RQPA plot of the sample obtained from 2.8Ti/2C/1.2TiS₂ mixture heated at 1100 °C for 3 min.

The refined synthesis parameters are concluded as follows: raw materials of Ti, TiS₂, and C with the molar ratio of 2.8:1.2:2, heated at 1100 °C for 3 min in argon atmosphere. Slightly excess of sulfur source in the starting reactant mixture could promote the productivity of Ti₂SC. It is believed that the excess of sulfur source could compensate the evaporation of sulfur.

3.2 Structure of samples of different temperatures

Figure 8 shows the SEM micrographs with EDS of selected areas of samples heated at 400–1100 °C. The atomic ratios of EDS analysis in areas marked in Fig. 8 are listed in Table 1. At 400 °C, the sample is composed of lamella structure and black block-shaped particles in the marked areas shown in Fig. 8(a), which is corresponding to a large amount of Ti₂SC and un-reacted graphite. In area I, the value of C is higher than the one in the formula which is in good agreement with the XRD results. When the temperature is in the range of 800 °C, the layered structure of Ti₂SC becomes more and more obvious, and the content of graphite reduces markedly. Finally, at 1100 °C, the ratio of Ti:C:S, in area III marked in Fig. 8(c), is consistent well with that of Ti₂SC, and the value of C is slightly higher

Table 1 EDS analysis of elements in samples of selected areas in Fig. 8

Selected area in Fig. 8	Ti atom ratio (%)	S atom ratio (%)	C atom ratio (%)	Corresponding phase
Area I	45.39	22.76	31.85	Ti ₂ SC, C
Area II	2.47	1.34	90.74	C, Ti ₂ SC
Area III	48.01	24.23	25.37	Ti ₂ SC

than the one in the formula which is in good agreement with the Rietveld quantitative phase analysis results. The layered structure of Ti₂SC particles is perfectly formed, and the crystal particle size approaches to homogeneity which is about 2–5 μm.

3.3 Proposed mechanism of Ti₂SC powder

The results of XRD and SEM indicate that the synthesis mechanism of Ti₂SC strongly depends on the starting materials. For the Ti/C/S system, the reaction at 1000 °C is expressed by the possible reaction:



At the temperature range of 1100–1200 °C, Ti₂SC phase increases with consuming TiC and TiS phases. It is noted that TiC and TiS are the prerequisite reactants of Ti₂SC, as shown in Eq. (3). This judgment is in good

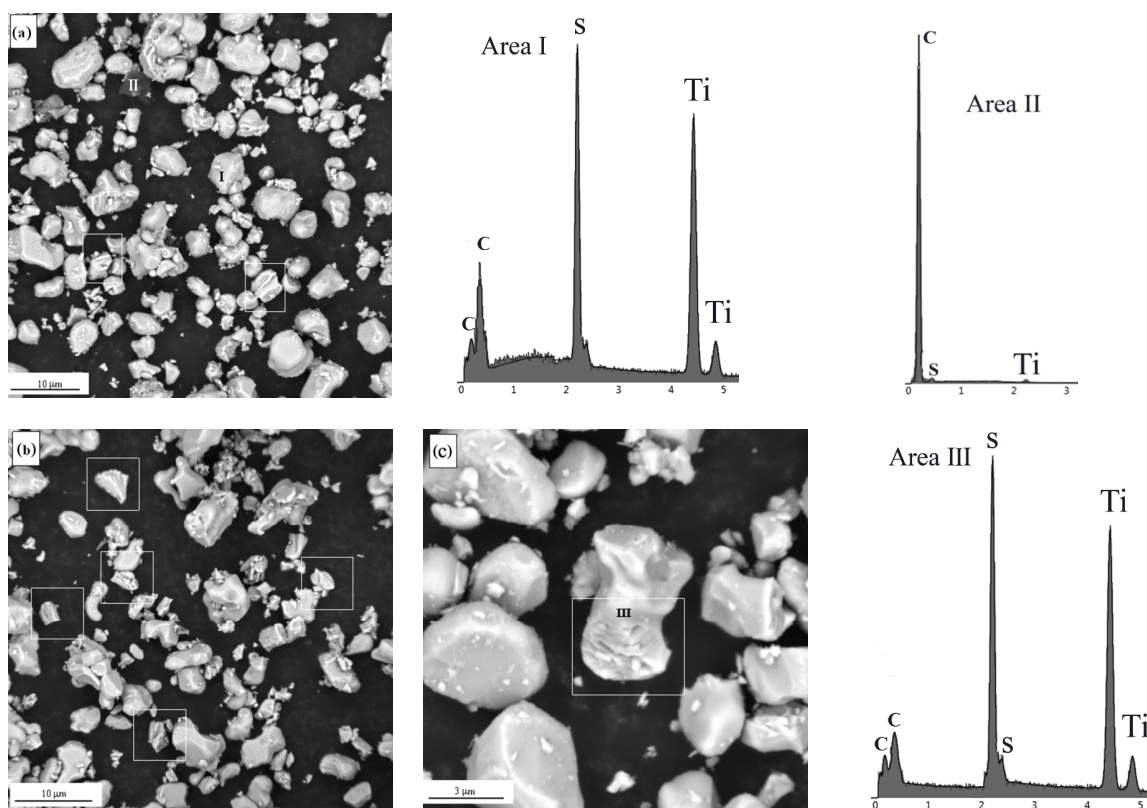
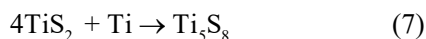
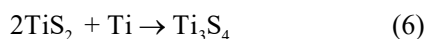
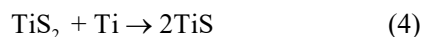


Fig. 8 Micrographs of SEM with EDS of 2.8Ti/2C/1.2TiS₂ mixtures heated at (a) 400 °C, (b) 800 °C, (c) 1100 °C.

agreement with another research [20].

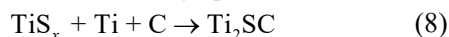


On the other hand, in the Ti/C/TiS₂ system, the TiS_x compounds (including Ti₅S₈, Ti₃S₄, and TiS) formed at 300 °C are concluded as follows:



According to the thermodynamic data [28], the changes in Gibbs free energy of the possible reactions of formed compounds are calculated as estimated at different temperature shown in Fig. 9. It can be seen that there are two possible reaction paths Eqs. (4) and (5) to form TiS, and the reaction Eq. (4) is thermodynamically favorable ($\Delta G < 0$), confirming the feasibility of proposed reaction, just the opposite of reaction Eq. (5). So Ti and TiS₂ are more possibly responsible for the generation of TiS. Because no data are available for Ti₃S₄ and Ti₅S₈, the stability is unable to predict using thermodynamic calculations. Actually, Ti₃S₄ and Ti₅S₈ are formed by the reaction of TiS₂ and Ti, which is proved by the XRD experiments. Based on these, the reactions (4), (6), and (7) are the paths to TiS_x compounds.

At 400 °C, it is noted that the amount of Ti₂SC increases significantly by consuming TiS_x (including Ti₅S₈, Ti₃S₄, TiS₂, TiS), Ti, and graphite:



There exist discrepancies in comparison with the product using Ti/C/S as raw materials which is observed TiS and TiC are the prerequisite reactants of Ti₂SC. However, the as-proposed reaction does not explain the reaction process in current microwave hybrid heating circumstance because of the absence of

TiC at low temperature.

On the basis of the present study, a possible mechanism is proposed to explain the synthesis of Ti₂SC in Ti/C/TiS₂ system at a temperature as low as 1100 °C, which is illustrated in Fig. 10. At the first stage, when the temperature is below 400 °C, the formation of the intermediate phase is mainly for the TiS_x intermetallic compounds (including Ti₅S₈, Ti₃S₄, and TiS). Meanwhile, un-reacted Ti and graphite are detected. At 400 °C, Ti₂SC begins to form by the reaction among TiS_x (including Ti₅S₈, Ti₃S₄, TiS₂, and TiS), un-reacted Ti, and graphite, and the amounts of TiS_x, Ti, and graphite notably decrease. As the sintering temperature continues to rise to 800 °C and above, high-purity Ti₂SC with a very small amount of TiS and graphite is fabricated. When the temperature increases to 1100 °C, Ti₂SC becomes the only phase in the sample, indicating all the reactions have been completed. By contrast with using S as sulfur source, the present synthesis temperature of Ti₂SC decreases by 400 °C and the yield of Ti₂SC improves considerably.

4 Conclusions

A large amount of Ti₂SC powder was synthesized at 800 °C in the Ti/C/TiS₂ system by microwave hybrid heating, whereas large amount of impurities was remained after heating at 1200 °C in the Ti/C/S system. Based on XRD, SEM, and thermodynamics, the difference of synthesis mechanism as the starting materials was identified. Introducing of microwave hybrid heating process decreased the synthesis temperature of Ti₂SC. For Ti/C/TiS₂ system, Ti₂SC could be synthesized at lower temperature than Ti/C/S system because TiS_x intermetallics reacted with Ti and

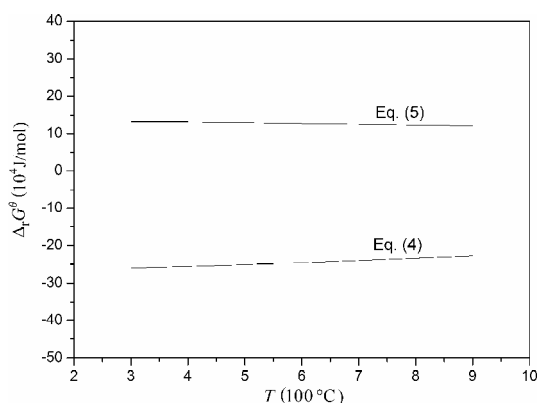


Fig. 9 Gibbs free energy of formation changed by temperature.

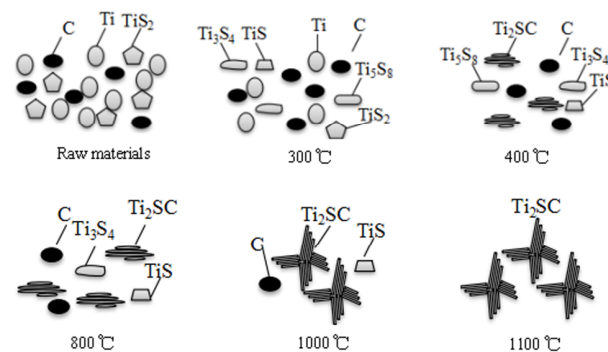


Fig. 10 Proposed formation mechanism of Ti₂SC in the Ti/C/TiS₂ system.

graphite to generate Ti_2SC at 400 °C under microwave hybrid heating circumstance. High-purity Ti_2SC was synthesized at 1100 °C without press by microwave hybrid heating using TiS_2 as sulfur source, and the particles of Ti_2SC powder were uniform and the average size was about 2–5 μm .

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